

# CEMENT AND LIME MANUFACTURE

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## New British Standard Specifications for Ordinary and Rapid-hardening Portland Cements.

THE British Standard Specification for Portland Cement, last issued in 1931, has been revised, and the new specification also deals with rapid-hardening Portland cement. The principal alterations in the new specification compared with the 1931 edition are: (1) the inclusion of rapid-hardening Portland cement with appropriate test figures; (2) the omission of the coarser sieve in the test for fineness of cement; (3) the substitution in the clause on chemical composition of a formula for the ratio of lime to silica and alumina in which weight percentages replace the previous chemical equivalents and allowance is also made for iron oxide; (4) the addition of a compression test on cement and sand mortar, for use when required by the purchaser; (5) the omission of the appendix relating to the optional tensile test on neat cement.

**Fineness.**—Samples of 100 grammes, when sifted continuously for fifteen minutes on a British standard sieve No. 170, shall have residues (by weight) of not more than 10 per cent. in the case of ordinary Portland cement or 5 per cent. in the case of rapid-hardening Portland cement.

**Chemical Composition.**—The percentage of lime, after deduction of that necessary to combine with the sulphuric anhydride present, shall be not more than 2.8 times the percentage of silica plus 1.2 times the percentage of alumina, plus 0.65 times the percentage of iron oxide, nor be less than two-thirds of that amount. The ratio of the percentage of iron oxide to that of alumina shall not exceed 1.5. The weight of insoluble residue shall not exceed 1.0 per cent., that of magnesia shall not exceed 4 per cent., and the total sulphur content calculated as sulphuric anhydride ( $\text{SO}_3$ ) shall not exceed 2.75 per cent. The total loss on ignition shall not exceed 3 per cent. for cement manufactured or sampled or tested in temperate climates and 4 per cent. for cement manufactured or sampled or tested in hot climates.

**Tensile Strength.**—The ultimate tensile strength of the briquettes made with 1 part of cement to 3 parts of standard sand is stipulated to be as follows :—

*Ordinary Portland Cement.*

3 days (72 hours).—Not less than 300 lb. per sq. in. (21.09 kg. per sq. cm.).

7 days.—An increase on the ultimate tensile stress at three days and not less than 375 lb. per sq. in. (26.37 kg. per sq. cm.).

*Rapid-hardening Portland Cement.*

1 day (24 hours).—Not less than 300 lb. per sq. in. (21.09 kg. per sq. cm.).

3 days (72 hours).—An increase on the ultimate tensile stress at one day and not less than 450 lb. per sq. in. (31.63 kg. per sq. cm.).

**Compressive Strength.**—At the option of the purchaser, a test for compressive strength may be substituted for the tensile test. For the compressive test, the cubes must have sides of 50 square centimetres (length of sides, 2.78 in.). Each cube must comprise 185 grammes of cement, 555 grammes of standard sand and 74 grammes of water, and the cubes are to be vibrated in a machine described in the specification. When the load is applied at the rate of 5,000 lb. per square inch per minute the cubes must have the following strengths :

*Ordinary Portland Cement.*

3 days (72 hours).—Not less than 1,600 lb. per sq. in. (112.5 kg. per sq. cm.).

7 days.—An increase on the compressive strength at three days and not less than 2,500 lb. per sq. in. (175.8 kg. per sq. cm.).

*Rapid-hardening Portland Cement.*

1 day (24 hours).—Not less than 1,600 lb. per sq. in. (112.5 kg. per sq. cm.).

3 days (72 hours).—An increase on the compressive strength at one day and not less than 3,500 lb. per sq. in. (246.1 kg. per sq. cm.).

Copies of the Specification (No. 12, 1940) may be obtained from the British Standards Institution (price 2s. net).

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## New United States Specifications for Portland Cement.

### FIVE TYPES OF PORTLAND CEMENT DESCRIBED.

THE American Society for Testing Materials has adopted new specifications for Portland cement, which became effective on September 2, 1940. The new specifications replace A.S.T.M. standard specifications for Portland cement (C9-38) and high-early strength Portland cement (C74-39), and also cover three other types of cement. The new specification is as follows.

#### Scope.

*Type I.*—For use in general concrete construction when the special properties specified for Types II, III, IV, and V are not required.

*Type II.*—For use in general concrete construction when the special properties specified for Types III, IV, and V are not required, and when the chemical and physical test requirements prescribed for this type in these specifications are desired.

*Type III.*—For use when high-early strength is required.

*Type IV.*—For use when a low heat of hydration is required.\*

*Type V.*—For use when high sulphate resistance is required.\*

#### Basis of Purchase.

The purchaser should specify the type or types desired. When no type is specified, the requirements of Type I shall govern.

#### Definition

*Portland Cement.*—Portland cement is the product obtained by pulverising clinker consisting essentially of hydraulic calcium silicates, to which no additions have been made subsequent to calcination other than water and/or untreated calcium sulphate, except that not to exceed 1 per cent. of other materials may be added, provided such materials have been shown not to be harmful by tests acceptable to Committee C-1 on Cement. *Note.*—Tests to determine whether a proposed addition is harmful will be carried out or reviewed by Committee C-1 on Cement, for those making requests, through its Cement Reference Laboratory or other laboratory which the committee may select. As such tests are completed or reviewed the committee will make known those additions which have been found not to be harmful.

#### Chemical Limits.

Portland cement of each of the five types shall conform to the requirements prescribed in *Table I*.

#### Physical Requirements.

Portland cement of each of the five types shall conform to the requirements prescribed in *Table II*.

\* Attention is called to the fact that cements conforming to the requirements for Type IV and Type V are not usually carried in stock. In advance of specifying their use purchasers or their representatives should determine whether these types of cement are, or can be made, available.

TABLE I  
CHEMICAL REQUIREMENTS

	Type I	Type II	Type III	Type IV	Type V
Magnesium oxide (MgO), max., per cent. . . . .	5.0	5.0	5.0	5.0	4.0
Sulphur trioxide (SO <sub>2</sub> ), max., per cent. . . . .	2.0	2.0	2.5	2.0	2.0
Loss on ignition, max., per cent. . . . .	3.0	3.0	3.0	2.3	3.0
Insoluble residue, max., per cent. . . . .	0.75	0.75	0.75	0.75	0.75
Silica (SiO <sub>2</sub> ), min., per cent. . . . .	—	21.0	—	—	24.0
Alumina (Al <sub>2</sub> O <sub>3</sub> ), max., per cent. . . . .	—	6.0	—	—	4.0
Iron oxide (Fe <sub>2</sub> O <sub>3</sub> ), max., per cent. . . . .	—	6.0	—	6.5	4.0
Ratio of Al <sub>2</sub> O <sub>3</sub> to Fe <sub>2</sub> O <sub>3</sub> . . . . .	—	0.7 to 2.0	—	—	0.7 to 2.0
Tricalcium silicate (3 CaO-SiO <sub>2</sub> ),* max., per cent. . . . .	—	50	—	35	—
Dicalcium silicate (2 CaO-SiO <sub>2</sub> ),* min., per cent. . . . .	—	—	—	40	—
Tricalcium aluminate (3 CaO-Al <sub>2</sub> O <sub>3</sub> ),* max., per cent. . . . .	—	8	15	7	5

### Packing and Marking.

When, as specified, the cement is delivered in packages, the name and brand of the manufacturer of the cement and the type shall be plainly identified. When, as specified, the cement is delivered in bulk, this information shall be contained in the advices accompanying the consignment. A bag shall contain 94 lb. net. A barrel shall consist of 376 lb. net. All packages shall be in good condition at the time of inspection.

### Storage.

The cement shall be stored in such a manner as to permit easy access for proper inspection and identification of each consignment, and in a suitable weathertight building that will protect the cement from dampness.

### Inspection.

(a) Every facility shall be provided the purchaser for careful sampling and inspection at either the mill or at the site of the work, as may be specified by the purchaser. The following periods from the time of testing shall be allowed for completion of testing: 1-day test, 6 days; 3-day test, 8 days; 7-day test, 12 days; 28-day test, 33 days.

(b) The purchaser shall have the right to observe the various operations connected with the manufacture of the cement and to keep such records thereof as he may desire.

\* The expressing of chemical limitations by means of calculated assumed compounds does not necessarily mean that the oxides are actually or entirely present as such compounds. The percentages of tricalcium silicate, dicalcium silicate, and tricalcium aluminate shall be calculated from the chemical analysis as follows:

$$\text{Tricalcium silicate} = (4.07 \times \text{per cent. CaO}) - (7.60 \times \text{per cent. SiO}_2) - (6.72 \times \text{per cent. Al}_2\text{O}_3) - (1.43 \times \text{per cent. Fe}_2\text{O}_3) - (2.85 \times \text{per cent. SO}_3)$$

$$\text{Dicalcium silicate} = (2.87 \times \text{per cent. SiO}_2) - (0.754 \times \text{per cent. 3 CaO-SiO}_2)$$

$$\text{Tricalcium aluminate} = (2.65 \times \text{per cent. Al}_2\text{O}_3) - (1.69 \times \text{per cent. Fe}_2\text{O}_3)$$

Oxide determinations calculated to the nearest 0.1 per cent. shall be used in the calculations. Compound percentages shall be calculated to the nearest 0.1 per cent. and reported to the nearest 1 per cent.

TABLE II  
PHYSICAL REQUIREMENTS

	Type I	Type II	Type III	Type IV	Type V
Fineness, specific surface, sq. cm. per g. :					
Average value, min. . . . .	1,600	1,700	—	1,800	1,800
Minimum value, any one sample . . . .	1,500	1,600	—	1,700	1,700
Soundness :					
Autoclave expansion, max., per cent. . .	0.50	0.50	0.50	0.50	0.50
Time of setting (alternative methods)					
Gillmore test :					
Initial set, min., not less than . . .	60	60	60	60	60
Final set, hr., not more than . . . .	10	10	10	10	10
Vicat test :					
Initial set, min., not less than . . .	45	45	45	45	45
Final set, hr., not more than . . . .	10	10	10	10	10
Tensile strength, lb. per sq. in. :†					
The average tensile strength of not less than three standard mortar briquettes composed of 1 part cement and 3 parts standard sand, by weight, shall be equal to or higher than the values specified for the ages indicated below :					
1 day in moist air . . . . .	—	—	275	—	—
1 day in moist air, 2 days in water . .	150	125	375	—	—
1 day in moist air, 6 days in water . .	275	250	—	175	175
1 day in moist air, 27 days in water . .	350	325	†	300	300
Compressive strength, lb. per sq. in. :†					
The average compressive strength of not less than three mortar cubes composed of 1 part cement and 2.75 parts fine testing sand, by weight, shall be equal to or higher than the values specified for the ages indicated below :					
1 day in moist air . . . . .	—	—	1,300	—	—
1 day in moist air, 2 days in water . .	1,000	750	3,000	—	—
1 day in moist air, 6 days in water . .	2,000	1,500	—	800	1,000
1 day in moist air, 27 days in water . .	3,000	3,000	†	2,000	2,200

### Rejection.

(a) The cement may be rejected if it fails to meet any of the requirements of these specifications.

(b) Cement remaining in bulk storage at the mill for a period greater than six months after completion of the tests may be retested and may be rejected if it fails to conform to any of the requirements of these specifications.

\* The purchaser should specify the type of setting time test required. In case he does not so specify, the requirement of the Gillmore test only shall govern.

† The purchaser should specify the type of strength test required. In case he does not so specify, the requirements of the tensile strength test only shall govern. The strength at any age shall be higher than the strength at the next preceding age. Tests at 28 days on Types I and II cement may be waived at the option of the purchaser. If, at the option of the purchaser, a 28-day test is required on Type III cement, the strength at 28 days shall be higher than at 3 days.

(c) Packages varying more than 5 per cent. from the specified weight may be rejected; and if the average weight, as shown by weighing 50 packages taken at random, is less than that specified, the entire consignment may be rejected.

#### Methods of Testing.

The cement shall be sampled and the properties enumerated in these specifications shall be determined in accordance with the following methods of the American Society for Testing Materials:

(a) *Chemical Analysis*.—Standard Methods of Chemical Analysis of Portland Cement (A.S.T.M. Designation: C 114-39)<sup>3</sup> and Tentative Methods of Chemical Analysis of Portland Cement (A.S.T.M. Designation: C 114-39 T).<sup>4</sup>

(b) *Sampling and Physical Tests*.—Standard Methods of Sampling and Physical Testing of Portland Cement (A.S.T.M. Designation: C 77).<sup>5</sup>

(c) *Compressive Strength*.—Tentative Method of Test for Compressive Strength of Portland Cement Mortars (A.S.T.M. Designation: C 109),<sup>6</sup> when specified.

(d) *Fineness*.—Tentative Method of Test for Fineness of Portland Cement by Means of the Turbidimeter (A.S.T.M. Designation: C 115).<sup>7</sup>

(e) *Autoclave Expansion*.—Tentative Method of Test for Autoclave Expansion of Portland Cement (A.S.T.M. Designation: C —).

Commenting on the new specifications, *Rock Products* says: "Since this is the first time an autoclave test has been required in any A.S.T.M. specification, the method of making the test is a matter of general interest. This has been published by the A.S.T.M. as information, but a year ago was rejected as a tentative standard method of test. The moulded specimens of neat cement bars are 1 in. by 1 in. by 10 in., with stainless steel or other non-corroding-metal reference points,  $\frac{1}{4}$  in. diameter. The distance between the reference points must be  $10 \pm 0.1$  in. These bars are put in an autoclave or boiler capable of raising the steam pressure to 295 lb. per sq. in. (gauge) or 310 absolute in from 1 to  $1\frac{1}{2}$  hours. This pressure is maintained for 3 hours and then reduced to 10 lb. pressure in 1 hour. After mixing and storage in the moulds in a moist room for at least 20 hours, the specimens may be removed from the moulds and similarly cured but must be put in the autoclave at 24 hours  $\pm$  30 minutes after making. The expansion in the autoclave must not be more than 0.5 per cent.

<sup>3</sup> 1939 Book of A.S.T.M. Standards, Part II, p. 8.

<sup>4</sup> *Ibid.*, p. 883.

<sup>5</sup> *Ibid.*, p. 17.

<sup>6</sup> 1939 Book of A.S.T.M. Standards, Part II, p. 892.

<sup>7</sup> 1939 Book of A.S.T.M. Standards, Part II, p. 897.

## Research on Portland Cement.

INTERESTING views on the chemistry and manufacture of Portland cement were given by Mr. P. H. Bates (of the United States National Bureau of Standards) in the Edgar Marburg Lecture delivered before the American Society for Testing Materials in June last. In the course of the lecture Mr. Bates said:

### Unknown Oxides.

The work done since Le Chatelier's time has confirmed most of his findings and extended them to give the proper place to the dicalcium silicate and the lime aluminoferrate, but has left the tricalcium aluminate and the flux still conspicuously uncertain. Uncertainty still remains as to the nature of the role of the so-called minor oxides. These include  $MgO$ ,  $Na_2O$ ,  $K_2O$ ,  $FeO$ ,  $TiO_2$ ,  $Mn_2O_3$ , and  $P_2O_5$ , which average a total of about 4 per cent., but in some cases amount to 6 per cent. They should be considered minor only because our knowledge of the nature of their existence is meagre. They constitute a total that should justify our being concerned about accepting the premise that they may be ignored (especially in studying the physical properties of cement).

Investigators have studied such ternary systems as  $SiO_2-Al_2O_3-CaO$ ;  $CaO-MgO-SiO_2$ ;  $CaO-FeO-SiO_2$ , etc., parts of the quaternary system  $CaO-SiO_2-Al_2O_3-Fe_2O_3$  and other oxide systems. Data concerning the phase equilibria in these have been obtained, and by means of such data it may be possible to predict the state of equilibrium in clinker, provided that among other criteria the conditions of heating and cooling and the maximum temperature of burning are known, and on the premise that all the lesser oxides in the mix have no effect upon the equilibria of the four major ones.

### Reactions with Water.

When Portland cement is mixed with water, it were better not to speak of hydrated cement, but rather of hydrating cement. The reaction is one which continues, if water can have access to all the grains, until the constituents are broken down into the hydrates of the oxides originally present (or carbonates of these if in some cases carbon dioxide is available). Then cementitious products no longer remain. Hydration extends from a minimum of no commercial significance through an optimum of greatest value to a maximum—again of no commercial value. Hence, all the physical properties must be defined as of a specific stated time.

It is possible to estimate approximately the heat of hydration of any cement from its chemical analysis. If the cement is high in alumina and lime and low in silica and iron oxide, it will contain predominantly the high-limed aluminates and silicates and will generate a large amount of heat. In large masses of concrete a high temperature will be reached at about the same time that rigidity will be attained. As the exterior cools the differences in volume between the exterior and the interior may cause cracking. Hence, if one wishes to remedy this condition one of the ways which suggests itself is through such chemical limitations as will bring about the presence of those constituents in the cement



as have low heat of hydration and rates of heat evolution. However, to be sure that this result has been obtained, the heats of hydration at certain specified ages should be determined by calorimetric methods.

There are many data of use in predicting the physical characteristics of a hydrated cement from a chemical analysis of the non-hydrated material. From such data we know that the lime aluminate or the lime aluminoferrate develops so little strength that the alumina and iron oxide may be ignored in this respect. The high-lime silicate, and consequently a high-lime low-silica cement, attains the greater part of its strength in a month; while the low-limed silicate, unless extremely finely ground and in the presence of other materials which act somewhat catalytically, at the same age has developed hardly a tenth of the strength of the other silicate. By the end of a year the strength of the two types of silicates shows practically no difference.

### The New Specifications.

Now that the American Society for Testing Materials is considering the acceptance of tentative specifications for five types of cement\* in which chemical requirements are so outstanding and physical requirements so dimly visible, it seems time to start active work in developing physical tests despite the visioned difficulties—and the difficulties are more of vision than of fact.

The relation between the calculated lime aluminate content of some cements and the expansion of test specimens made of these, subjected to alternations of freezing and thawing, was used in part in aiding in setting the limit on the permissible amounts of the calculated  $3\text{CaO} \cdot \text{Al}_2\text{O}_3$  in the proposed tentative specifications for type II cement. These data have been used to show: (1) There is a positive trend in the relation between the amount of this compound and the resistance to freezing and thawing; (2) there is a positive lack of trend in this relation; and (3) there is a trend sufficient to justify the use of the 8 per cent. limit presented in the new standard.

Let us put ourselves in the position of the manufacturers of two cements shown to have approximately 11 per cent. aluminate and to have withstood 175 cycles of freezing and thawing. Certainly we can rightly protest against the 8 per cent. limit since, although our products are 33 per cent. in excess of the permissible limit, they are better in the freezing-and-thawing tests than the majority of the cements within that limit. Then in our role of specification writer we would tell the producers that, granted that their contention is correct, the large majority of the cements exceeding the limit showed a much more unfavourable deportment in the physical test than those within the limit. Further, we cannot adopt limits which will permit a few exceptional cements to be accepted when by so doing we allow the use of a much larger number of inferior ones. But in a third role—that of an investigator—we say that something is wrong if we hold (as we must) that the only variable in the specimens was the aluminate content of the cements and that all specimens were similarly tested. In the role of producer we would demand to be shown that our cement contained 11 per cent.

\* See p. 173 of this number.



aluminate; we would not accept calculated values. Finally, as a specification writer, we would have to confess that we have no way of meeting such demands.

### Raw Materials.

The availability of raw materials and the possibility of commercially securing the ranges in composition that will yield cements meeting one or more of the five sets of requirements demand some attention. In order to make all five types most manufacturers will have to bring to their plants varying amounts of raw material now not at hand. But this does not necessarily mean that at all times these compositions will yield the values demanded by the physical tests. Neither does it mean that other compositions will not at times give the required physical results and hoped-for job results. It does, however, mean that at the present stage we have not either completely correlated composition and physical properties or completely correlated chemical composition and all the compounds actually present in the cement. However, the specifications must base their requirements, within limits, on the deportment of the majority rather than on the exceptional minority, even though some of the minority in certain respects may be the more desirable. In certain special cases, the choice of the ideal exception should apply.

There may be some question in the mind of the user as to the choice of types I or II. In an increasing number of cases the type I made by a considerable number of producers is identical with their type II, and this number will increase with time. Types IV and V are for use when low heat of hydration and sulphate resistance respectively are of importance; they will not generally be kept in stock by producers, at least for some time.

### Fineness of Product.

It would seem that within reasonable limits the manufacturer should be allowed to grind to whatever fineness yields the specified physical properties that are affected by the degree of comminution. The physical properties mostly affected by fineness are time of set and strength. Thus, a low-heat cement can be brought into the class of type I cement by grinding alone. The only distinction between many high-early-strength cements and type I cement is that the producer has taken some of his type I clinker and ground it about 1,000 sq. cm. per gram finer than he would if making type I cement.

It is not necessary to grind all kinds of clinker to the same degree of fineness to obtain the same strength. Why, therefore, not allow the producer to use whatever fineness will yield the strength indicated for each type? Why compel all to grind to the same minimum fineness? The answer is that many believe that fineness is closely associated with other properties, mostly undesirable, which we cannot cover by test methods or limitations.

Even under the high limits for the autoclave expansion test (0.5 per cent.) specified the user is given additional protection so far as quality is concerned. Few producers have any difficulty in making "autoclave cement" when using the compositions required by types II, IV and V. The low-heat cement purchased by the Bureau of Reclamation must not exceed a maximum expansion of

0.2 per cent. according to this test. This is 60 per cent. less than that allowed by the Society's new tentative specifications. Some producers had difficulties in meeting the demands when making cements of types I and III, but they have learned that, among other things, more rapid cooling of the clinker will result in a marked decrease of expansion in the high-pressure steam testing in the autoclave.

#### **Admixtures.**

The Society's standards, first for high-early-strength cement and in 1938 for Portland cement, were amended so that under certain conditions admixtures are permitted. There is now an agitation to permit the use of other admixtures. Some of these have been found advantageous in increasing the life of concrete in highways subjected to sanding with calcium chloride during freezing conditions. Most of these are of such a nature that frothing takes place during mixing, and consequently as much as 10 per cent. or more of air may be introduced into the wet concrete. This gives a readily placeable mix, which before setting releases water to a negligible degree. This admixture is used in very small quantities—mostly less than 0.05 per cent. of the weight of the cement.

There is no reason to assume that improvement in other physical properties cannot be secured by means of other additions. The setting and hardening of cement is a chemical reaction, and there are many examples of the marked alterations in reactions induced by the presence of small quantities of foreign materials. At times such reactions may be adversely affected by the small amounts of additions—as in the slowing of setting and hardening induced by very small quantities of solutions of sucrose or lead or zinc salts. But such positive results even though adverse should only seem to confirm the thought that there must be other materials which can cause most advantageous effects. We know that the rate of the reaction can be materially accelerated by fine grinding, by heat, and to a less degree by the use of calcium chloride, etc. There is likely a host of other materials which will speed up the reaction, possibly to the extent that within a day we may have the strength usually attained at the end of a year. It is curious that so much time has been spent in developing different kinds of cement and so little on developing very promptly by means of admixtures the full inherent qualities of the cements already at hand.

At this time there is a marked tendency to depart from the high-lime cements to the high-silica ones. These, like the low-heat and sulphate-resisting types, unless very finely ground, are also slow hardening. But if this characteristic could be overcome through the use of an admixture acting as an accelerator we would have approached very closely to the ideal all-purpose cement. There is not much doubt that this accelerator will be found some day—even though it may not for psychological reasons be called an admixture.

#### **Manufacturing Processes.**

Furnace design has materially developed since the advent of the rotary kiln. Even the old stack kiln could be slightly modified to permit of more uniform operation and control than the rotary kiln. The installation of regenerators or

recuperators would reduce it to a very low-cost fuel furnace. But the ceramic industry, which has so long favoured the stack kiln, is supplanting it with tunnel or ring kilns, due to their increased fuel economy and ease of control. Either of these two types would permit of wider ranges of rates of heating and cooling, and the ring type would permit of simultaneous burning of several types of cement requiring different burning temperatures.

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## Calcining Portland Cement.

In the course of a review of the present knowledge of burning Portland cement, published in the May, 1940, number of *Revue des Matériaux de Construction*, M. H. E. W. Lutz states :

The reactions in the burning of cement have been the special study of Dyckerhoff and Prüssing. The latter has sought to ascertain what happens during the successive stages of burning by systematic studies of samples drawn from the different zones of a wet-process rotary kiln. To understand the process of burning, he determined the calcium carbonate content, calculated as CaO, the free-lime content, and the insoluble silica content in the kiln charge. Allowing for the possible presence of soluble silica and combined calcium oxide, he arrived at the following conclusions. A reaction between the calcium carbonate and the clay takes place before the formation of free lime. Between 900 deg. C. and 950 deg. C., after about one-half of the calcium carbonate, calculated as oxide, had reacted, the free-lime content increased rapidly. At about 20 ft. from the wet end of a rotary kiln about 60 ft. long, the reaction between the chalk and the clay becomes marked; it is there that the cement compounds are first formed. The free-lime and insoluble silica contents of the mix are quickly reduced in quantity, and at the same time the proportion of combined lime is increased in the new compounds being formed. The reactions here (1,000 deg. C. to 1,250 deg. C.) are exothermic.

According to Dyckerhoff, the attainment of phase equilibrium is completely dependent on the temperature and duration of burning. In the burning of clinker in a rotary kiln equilibrium is scarcely reached at 1,450 deg. C., at which temperature are formed dicalcium and tricalcium silicates together with a little free lime in the solid state. On cooling, brownmillerite ( $C_4AF$ ) and tricalcium aluminate crystallise from the liquid phase. Even when equilibrium between the solid and liquid phases is reached at 1,450 deg. C. it is not maintained on cooling; tricalcium aluminate crystallises out and brownmillerite separates from the liquid, a portion of the latter solidifying as glass.

Bogue maintains that it is possible, using a phase equilibrium diagram, to calculate the compounds in a clinker for any given analysis of the raw material used in the manufacture of the clinker. It is not, however, possible to calculate the glass content in a clinker. This is also true for the method of calculation proposed by Lea and Parker. If it is considered that in the formation of clinker

minerals during burning there is an obvious "non-equilibrium" condition, one is led to think that such methods of calculation are only of limited value.

The process of burning is best judged by the inspection and supervision of the clinkering; by control of the composition and temperature of the kiln gases; by watching the colour and hardness of the clinker; and by laboratory testing of the cement for constancy of volume, strength, and chemical composition. Well-burned cements are generally greenish-black or steel-blue in colour. Clinker burned in a reducing flame is usually brown, the brown core of the pieces being sometimes surrounded with a black covering.

Provided that the chemical composition is normal it may happen that a clinker is not sound. The cause may be that iron is present as  $\text{FeO}$  and has not been combined in the clinker minerals; it then follows that the ratio of calcium oxide to the sum of the iron oxide, alumina, and silica, i.e., the hydraulic modulus, is so large that a larger amount of free lime is formed than would have been the case under normal conditions.

An underburned clinker has a bad colour and gives low strength; probably the strength is unfavourably influenced by a particularly high fraction of non-crystallised glass. In addition, in a similar clinker, part of the iron oxide, because of the thermal dissociation of  $\text{Fe}_2\text{O}_3$  to  $\text{FeO}$  and  $\text{O}_2$ , may not have taken part in the formation of the clinker compounds. The determination of the loss on ignition, the free-lime content and insoluble silica content gives a useful relative indication of the burning conditions.

One method for the quantitative determination, by means of the polarising microscope, of the constituent minerals in clinker has been recently described by Schwiete. In the present state of its development, however, it does not appear suitable for use under manufacturing conditions. Along with this method, mention should be made of Anselm's method for determining the degree of burning of clinker. This consists of the rapid determination of the weight per litre of a fraction of the clinker; for example, a fraction made up of pieces of from 5 mm. to 7 mm. ought to represent, with respect to its cementing properties, the mean value for the whole clinker. This method has been checked by several other workers, notably Haegermann, Mussgnung, Würzner, and Lutz, all of whom have concluded that it can only be applied where an approximate idea of the quality of clinker is required.

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## Water Required in Wet Process Reduced by Slurry Filters.

WE take the following from a discussion on the advantages of the use of slurry filters which appeared in a recent number of the Cement Mill Edition of *Concrete* (June, 1940).

Prior to the introduction of the slurry filter in cement manufacturing, considerable attention had been given to methods of reducing the quantity of water required in the wet grinding process. Unfiltered slurry, when entering the kiln, may contain between 35 and 50 per cent. of its weight in water, at least 35 per cent. of which was necessary to obtain the fluidity required for the slurry to flow through the feed line to the kiln. This condition required the evaporation of from 200 lb. to 300 lb. of water, or more, per barrel of clinker. The complete evaporation of this water within the kiln involved, in extreme cases, the removal of approximately 325 lb. of water per barrel of clinker, which required about 17 lb. of coal.

By passing the wet raw material through slurry filters before entering the kiln, it is possible to reduce the water content as low as 18 per cent. or less. Instances can be cited where the reduction of water content gained through the installation of slurry filters ranges from 34 to 17 per cent., from 38 to 22 per cent., and from 48 to 29 per cent. In the first instance the raw materials were limestone and slag; in the second instance, limestone and clay; and in the third instance, chalk and clay. A study was made in 1930 of nineteen cement factories in which slurry filters were used. Reduction of moisture content as the result of filtration ranged from 50 to 60 per cent., and the saving in coal ranged from 25 lb. to 45 lb. per barrel of cement.

Saving in fuel is one of the three major advantages. The other two are, (1) an increase in output; and (2) an improvement in the quality of the finished product. In the nineteen works mentioned, the investigation disclosed that the capacity of the kilns was increased from 20 to 40 per cent. In cases where the water was purchased, the reduction in moisture content permitted the re-use of the water, and further reduced the cost. In the matter of improved quality, the clinker is more uniform and more easily ground after the slurry filters are installed.

### Types of Filters.

Very little additional labour is required to operate slurry filters, and the maintenance cost is low. It is estimated that  $1\frac{1}{2}$  cents per barrel of cement usually covers the cost of maintenance, the cost of additional power, and the cost of replacement of parts in the filters. The original cost of installation is not large.

In general, two types of slurry filters are employed, namely, the drum type and the disc type (*Fig. 1*). In the drum type the cycle of operations is as follows:

The drum, covered with a filter medium, is mounted on a horizontal axis within a tank. The tank contains the slurry from which a portion of the water content is to be removed. The level of slurry in the tank is kept constant. The drum is partially immersed in the slurry and revolves slowly through it. The surface of the drum is divided into shallow compartments, individually connected by pipes to an automatic valve mounted on one of the supporting trunnions. Each compartment, as it enters the slurry, is subjected to a vacuum controlled by the automatic valve, and a cake of the solid particles in the slurry is formed on the filter medium. The filtered liquid passes through this cake and filter compartments and through the internal connections to the valve. When the cake emerges from the slurry it is still under vacuum, which dries it by removing the entrained moisture and drawing in air.

The disc filter uses the same principle, but it provides a comparatively greater exposed surface for the action of the filtering and drying processes. The filtering

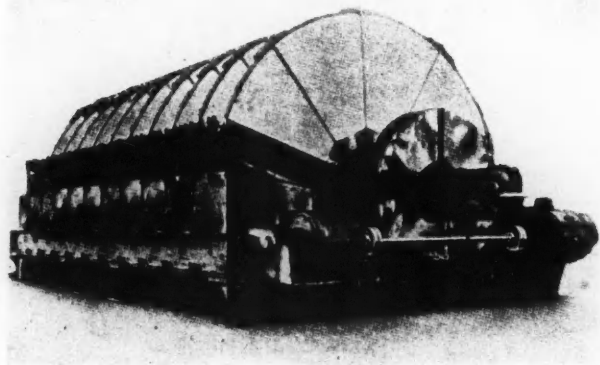


Fig. 1.—Disc Type Filter.

medium is attached to these discs, which are built up of wedge-shape sectors carried on a hollow shaft mounted in a crenellated tank. The hollow space within the shaft is divided into compartments. Each wedge-shape sector of the disc is constructed of a grooved or metal frame covered with a suitable filter medium and made so as to be readily attached to or removed from the central shaft. The discs, held under vacuum, are revolved slowly through the slurry in the tank, and a cake of solid particles is picked up on each side. Again the filtrate passes through the filter medium, along the drainage channels, into the hollow shaft, and then through a valve similar to that used on the drum filters.

Summarising the advantages of filtration of slurry, the first may be stated as that of reducing the quantity of water, and consequently reducing the fuel requirements. A complementary advantage lies in the fact that in the wet



grinding operations all the water needed may be used ; no consideration need be given to reducing the quantity of water in the slurry, inasmuch as the excess will be removed by the filters. This permits finer grinding through the use of the optimum quantity of water. Mud rings within the kilns are largely eliminated by the use of filters. Reduction in fuel consumption may be as high as 25 per cent. of the quantity used prior to the installation of the filters. An interesting point is that little change has been noted in the last eight or ten years. This may at first seem surprising when it is remembered that the first installation was made only fifteen years ago ; the reason is that filtration had been highly developed before it was introduced into cement manufacture.

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## Fine Grinding and Air Separation.

AN account of the grinding plant, specially designed for the manufacture of rapid-hardening Portland cement, at the works in Pennsylvania of the Nazareth Cement Co. is given in the June, 1940, number of *Rock Products*. The following is an abstract of the article, which was written by Mr. B. Nordberg.

### Preliminary Grinding.

Preliminary grinding of clinker, for all cements, is done with Hercules mills closed-circuited with Hum-mer vibrating screens. The clinker is very hard burned, and is cooled to an average temperature of about 150 deg. F. before grinding. Gypsum and clinker are proportioned by weight into two mill bins, and a 2½-mesh internal screen is used when grinding cements of ordinary fineness. Using this screen opening, the mill product averages 22.7 per cent. minus 200-mesh ; 28.8 per cent. through 100-mesh, 36.6 per cent. passing the 50-mesh, and 55.5 per cent. through the 20-mesh sieve. After being elevated and put over eight 4-ft. by 5-ft. Tyler screens having 10-mesh wire, the material passing through has the following analysis : 37.7 per cent. minus 200-mesh ; 47.3 per cent. minus 100-mesh ; 59.4 per cent. minus 50-mesh ; and 86 per cent. minus 20-mesh. This is the feed for the tube mills. Of the tailings, 93.5 per cent. is minus 6-mesh ; 49.3 per cent. minus 10-mesh ; 8.6 per cent. minus 14-mesh ; 2.8 per cent. minus 20-mesh ; and nothing minus 50-mesh. Tailings are returned to the same mills from which they were discharged. The circulating load between the screens and mills is 75 to 80 per cent. and the output is 150 barrels per mill per hour.

### Tube Mills in Open Circuit for Standard Cement.

The feed for the tube mills from the screens is conveyed by a drag chain and divided into eight tube-mill hoppers from which the feed is introduced axially through trunnion (screw) feeders. The trunnion feeders are driven through gear drives, with a 4 : 1 ratio of speed variation, which are control units that contribute





greatly toward the flexibility in operation of the mills when manufacturing several cements. Eight 5½-ft. by 20-ft. tube mills, in open circuit, can grind more material than is produced by the preliminary mills. Standard Portland cement, at 1,600 sq. cm. per gram or 92 per cent. through a 200-mesh sieve, is ground at the rate of 45 barrels per mill hour. The mills contain 20 tons of ¾-in. to 1-in. steel balls.

In making cements of ordinary fineness, the tube-mill discharges are collected into a hopper over an 8-in. Fuller-Kinyon pump, and are pumped into the stockhouses. Over the pump is a 4-ft. by 5-ft. screen with 2½-in. rectangular openings to protect it from receiving extraneous material from the mills. Cements of special compositions, such as high-silica cement, high-iron cement, modified cement, and cement of unusual colour are clinkered and ground either in open or closed circuit. The drive on each tube mill (peripheral discharge) is changed to open or closed circuit grinding by simply resetting an indicator at a predetermined point to slow up or speed up the screw feed.

Five hours are needed to change the mill over from standard grinding to fine grinding with air separation, and the reverse. Changes are made in the screen openings within the preliminary mills and on the screens, and feed spouts or tailings returned to the tube mills are connected.

#### Fine Grinding with Air Separation.

An 8-mesh screen opening within the preliminary mills is substituted on very fine grinding, and a 40-mesh wire is used on the screens. The mill discharge comprises about 58.2 per cent. minus 200-mesh; 69 per cent. minus 100-mesh; 83 per cent. minus 50-mesh; and 99.5 per cent. minus 20-mesh. It has a surface of about 729 sq. cm. per gram containing about 42.9 per cent. minus 325-mesh. The distribution of particle sizes according to microns is as follows:

Per cent.	Microns
8.4 .. ..	0 to 7½
10.8 .. ..	7½ to 10
15.8 .. ..	10 to 15
20.9 .. ..	15 to 20
25.1 .. ..	20 to 25

Tailings, which return to the preliminary mills (100 per cent. circulating load), comprise 5.7 per cent. minus 200-mesh; 7.2 per cent. minus 100-mesh; 14.2 per cent. minus 50-mesh; 87.8 per cent. minus 20-mesh; and 98.2 per cent. minus 14-mesh. The fines (tube mills feed material) average 66.7 per cent. through 200-mesh; 78 per cent. minus 100-mesh; and 91.7 per cent. minus 50-mesh, with a trace minus 20-mesh. The surface is about 942 sq. cm. per gram, or 58.1 per cent. minus 325-mesh. Size distribution is as follows:

Per cent.	Microns
9.9 .. ..	0 to 7½
13.6 .. ..	7½ to 10
20.9 .. ..	10 to 15
27.8 .. ..	15 to 20
33.3 .. ..	20 to 25

Handling of the tube-mill feed material is done in the same way as for standard cement. The product is pulverised to about 2,800 sq. cm. per gram, with an output of 18 to 20 barrels of finished cement per hour per mill. This compares with 10 to 12 barrels in open circuit, without air separation and with water-sprayed tube mills.

Tube mills are closed-circuited with four 14-ft. Sturtevant air separators. The mill discharges are carried by screw conveyor to a hopper feeding a 10-in. Fuller-Kinyon low pressure pump, which pumps the material into the air separators.

A surge tank over the air separators, which are arranged close together, receives the pipeline discharge, and the load is first divided to two pairs of separators and is then re-divided by a slide in a connecting screw conveyor to each separator. The ammeter readings on the air separator drives and other checks serve to control the extent of the feed into each separator.

The air separator fines are pumped into the stockhouse by an 8-in. pump, and the tailings are also handled by pump. At first the tails were conveyed into the drag chain conveyor carrying the material passing the screens to the tube mill hoppers. This was not satisfactory, as it caused inconsistencies in grinding performance and adversely affected grindability. Present practice is to pump the separator tailings into an independent screw conveyor, dividing them into separate overhead hoppers from which they are fed into the mills separately from the fresh feed material. When making high-early-strength cement, sections of pipe are connected from the tailing conveyor to each trunnion feeder, and the tails and fresh material are intimately mixed in definite proportions. In this way, the percentage of tailings to fresh feed is held constant in all mills. The circulating load is kept at about 146 per cent.

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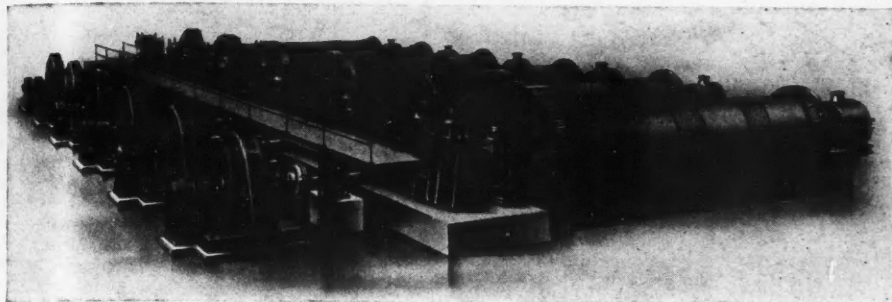
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★

(2) More Groups and bigger Savings are needed to make sure of victory. Go to it.

★

(3) There are only a few thousand groups to meet the needs of more than 2,000,000 employees. It is up to you to improve the position in your own industry.

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(4) There is a scheme to suit your requirements. Get in touch with your Local Savings Committee at once. Delay is helping the enemy.

★

(5) Form a Group in your firm, and play your own part with the thousands of other employers who have already taken this essential step towards victory.

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- (2) That firms which already have a Savings Group in existence should stimulate its membership and increase the rate of its subscriptions by arranging for:—

A meeting to be addressed by representatives of the firm, by responsible Trade Union officials or officers of the National Savings Movement.

An appeal to be sent to members urging increased support for the Group, the display of posters, or the distribution of leaflets which may be obtained from the National Savings Committee.

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